

FTA0407 10/11

(11) (A) NO. 1 124 487

(45) ISSUED 820601

(52) CLASS 23-341

(51) INT. CL. B01D 53/34³

(19) (CA) **CANADIAN PATENT** (12)

(54) PROCESS FOR SELECTIVE REMOVAL OF CYCLIC
UREA FROM HINDERED AMINE GAS TREATING
SOLUTION

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(73) Granted to Exxon Research and Engineering Company
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(21) APPLICATION NO. 342,448

(22) FILED 791221

(30) PRIORITY DATE U.S.A. (972,500) 781222

NO. OF CLAIMS 15

- 1 -

1 BACKGROUND OF THE INVENTION

2 FIELD OF THE INVENTION

3 This invention relates to a process for the removal
4 of a cyclic urea reaction product in an amine gas treating
5 process which involves an absorption and a regeneration sys-
6 tem. More particularly, the invention is concerned with the
7 selective precipitation and filtration of a cyclic urea
8 degradation product which forms as a by-product in the feed
9 gas scrubbing process.

10 DESCRIPTION OF THE PRIOR ART

11 It is well known in the art to treat gases and
12 liquids, such as mixtures containing acidic gases including
13 CO_2 , H_2S , SO_2 , SO_3 , CS_2 , HCN , COS and oxygen and sulfur
14 derivatives of $\text{C}_1 - \text{C}_4$ hydrocarbons with amine solutions to
15 remove these acidic gases. The amine usually contacts the
16 acidic gases and liquids as an aqueous solution containing
17 the amine in an absorber tower with the aqueous amine solution
18 contacting the acidic fluid countercurrently.

19 The acidic scrubbing processes known in the art can
20 be generally classified into three (3) categories.

21 The first category is generally referred to as the
22 aqueous amine process where relatively large amounts of amine
23 solution are employed during the absorption. This type of
24 process is often utilized in the manufacture of H_2 for ammonia
25 production where nearly complete removal of the acid gas, such
26 as CO_2 is required. It is also used in those instances where
27 an acid gas, such as CO_2 occurs with other acid gases or
28 where the partial pressure of the CO_2 and other gases are low.



1 The second category is generally referred to as the
2 aqueous base scrubbing process or "hot potash" process. In
3 this type of process a small level of an amine is included as
4 an activator for the aqueous base used in the scrubbing solu-
5 tion. This type of process is generally used where bulk
6 removal of an acid gas, such as CO₂ is desired. This process
7 also applies to situations where the CO₂ and feed gas pres-
8 sures are high. In such processes, useful results are
9 achieved using aqueous potassium carbonate solutions as amine
10 activators.

11 A third category is generally referred to as the
12 non-aqueous solvents process. In this process, water is a
13 minor constituent of the scrubbing solution and the amine is
14 dissolved in the liquid phase containing the solvent. In
15 this process up to 50% of amine is dissolved in the liquid
16 phase. This type of process is utilized for specialized
17 applications where the partial pressure of CO₂ is extremely
18 high and/or where many acid gases are present, e.g., COS,
19 CH₃SH, and CS₂.

20 The present invention relates to a process for the
21 selective separation of a cyclic urea degradation product
22 which may form as a by-product of the practice of the second
23 category of acid scrubbing process described above, namely,
24 the aqueous base scrubbing process or "hot potash" process
25 in which a hindered amine is used.

26 Many industrial processes for removal of acid gases,
27 such as CO₂, use regenerable aqueous alkali scrubbing solu-
28 tions, such as an amine and potassium carbonate which are
29 continuously circulated between an absorption zone where
30 acid gases are absorbed and a regeneration zone where they
31 are desorbed, usually by steam-stripping. The capital cost
32 of these acid scrubbing processes is generally controlled by
33 the size of the absorption and regeneration towers, the size
34 of the reboilers for generating stripping steam, and the size
35 of the condensers, which condense spent stripping steam so
36 that condensate may be returned to the system to maintain
37 proper water balance. The cost of operating such scrubbing

1 plants is generally related to the amount of heat required
2 for the removal of a given amount of acid gas, e.g., thermal
3 efficiency, sometimes expressed as cubic feet of acid gas
4 removed per pound of steam consumed. Means for reducing the
5 costs in operating these industrial processes have focused
6 on the use of absorbing systems or combinations of chemical
7 absorbants which will operate more efficiently and effectively
8 in acid gas scrubbing processes using existing equipment.

9 It is disclosed in U.S. Patent Nos. 4,112,050;
10 4,112,051 and 4,112,052 that sterically hindered amines
11 unexpectedly improve the efficiency, effectiveness and cyclic
12 working capacity of the acid gas scrubbing processes in all
13 three of the above-mentioned process categories. In the
14 case of the sterically hindered amine activated "hot potash"
15 CO₂ containing acid gas scrubbing process of the invention
16 described in U.S. Patent No. 4,112,050, the process can be
17 operated at a cyclic working capacity significantly greater
18 than when diethanolamine or 1,6-hexanediamine is the amine
19 activator used in a similar process. It is postulated that
20 the increase in cyclic capacity observed with the sterically
21 hindered amines is due to the instability of their carbamates.
22 In that respect, sterically hindered amines are similar to
23 tertiary amines. Tertiary amines are not used on a commer-
24 cial scale for carbon dioxide containing acid gas scrubbing
25 due to their low rates of absorption and desorption.

26 N-alkyl alkylene diamines are advantageously used
27 as sterically hindered amine activators in the "hot pot"
28 process. A preferred sterically hindered amine used as an
29 activator in the "hot pot" process is N-cyclohexyl-1,3-
30 propanediamine. This amine in the presence of an amino acid
31 is sufficiently water soluble under absorption and desorption
32 conditions to maintain a single phase and it also has a very
33 high absorption capacity.

34 Although N-cyclohexyl-1,3-propane diamine has been
35 found to produce excellent results as an activator in the "hot
36 pot" treating process, one drawback in processes where it has

1 been used is that it produces a cyclic urea product when the
2 acid treated gas is rich with CO₂ and also contains H₂S. The
3 cyclic urea has a deleterious effect on CO₂ removal rates and
4 must be removed and replaced with fresh N-cyclohexyl-1,3-
5 propanediamine. The makeup rate for the hindered amine has
6 a minimal effect on the process economics; however, the cyclic
7 urea that is formed must be selectively removed in order to
8 be able to maintain acid gas removal performance.

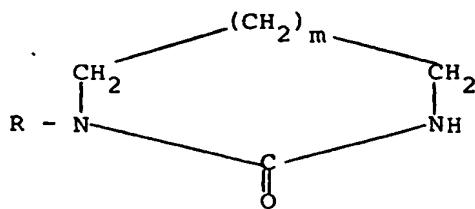
9 The invention which is disclosed herein represents
10 an improvement to the "hot pot" amine activated gas treating
11 process which includes the use of a hindered amine having a
12 tendency to form cyclic ureas under CO₂ rich conditions in
13 the presence of H₂S. This invention discloses a processing step
14 wherein the cyclic urea can be selectively removed from the
15 circulating solution thereby preventing any loss in acid gas
16 removal capabilities.

17 SUMMARY OF THE INVENTION

18 An acid gas scrubbing process providing for the
19 selective separation of a cyclic urea reaction product which
20 forms as a by-product during the acid gas removal, said pro-
21 cess comprising:

22 (a) contacting an acid gas mixture with an aqueous
23 solution, preferably in countercurrent flow, in an absorption
24 zone, said aqueous solution comprising an alkaline material
25 comprised of a basic alkali salt or metal hydroxide selected
26 from the group consisting of alkali metal bicarbonates, carbon-
27 ates, hydroxides, borates, phosphates and their mixtures, and
28 an activator for said basic salt comprising at least one
29 sterically hindered amine having the generic formula:

30 R-NH-(CH₂)_m-NH₂
31 where R is a secondary or tertiary alkyl or cycloalkyl hydro-
32 carbon having 4-20 carbon atoms and m is 2-5, at elevated
33 temperatures and pressures such that a cyclic urea degrada-
34 tion product having the generic formula:



where R is a secondary or tertiary alkyl or cycloalkyl having 4-20 carbon atoms and m is 0-3; is formed and a preferred loading of about 1 to about 10 SCF of acid gas per gallon of said aqueous solution is achieved;

(b) passing the acid gas rich aqueous solution produced from said step (a) to a regeneration zone preferably operated at temperatures ranging from about 200⁰F to about 250⁰F and pressures ranging from about 1 psig to about 15 psig where it is contacted, preferably in countercurrent flow, with steam to strip the acid gas impurities therefrom;

(c) cooling a portion of the lean solution exiting from said regeneration zone to temperatures ranging from about 80-180° F such that said cyclic urea degradation product is selectively precipitated from said lean solution;

(d) passing said lean solution containing said precipitated cyclic urea degradation product to a separation zone to remove at least a portion of said cyclic urea degradation product from said lean solution.

In a preferred mode of operation, the invention comprises the additional steps of:

(e) monitoring the accumulation of said degradation product in said separation zone, preferably a filter medium, until the pressure drop across said filter medium reaches about 25 psi

at which time said filter is segregated and a clean filter is substituted therefor;

(f) the segregated filter is cleaned by washing with hot water having a preferred temperature from about 200°-240°F.

The degradation product forms primarily at rich conditions and gradually builds up in the solution causing a drop-off in acid gas removal capabilities for the circulat-

1 ing solution. When there is a buildup, it becomes necessary
2 to remove the cyclic urea degradation product so as to main-
3 tain the enhanced acid gas removal rate for the hindered
4 amine solution. This is accomplished in the process of the
5 present invention by providing a slip stream from the lean
6 solution which is cooled to a preferred temperature of 130°-
7 160°F and then passed over a bed of carbon or a filter medium.

8 The cyclic urea material was found to have unanti-
9 cipated solubility properties when present in the circulating
10 solution which contains K_2CO_3 , $KHCO_3$, KHS, the sterically
11 hindered amine and amino acid. The cyclic urea which has
12 certain physical properties similar to those of the hindered
13 amine was found unexpectedly to selectively come out of the
14 solution as it was cooled from 200°F to a temperature of about
15 180-80°F. The desired specific temperature may be chosen on
16 the basis of the concentration of K_2CO_3 in the solution. In
17 this connection a typical temperature operating range for
18 various K_2CO_3 concentrations is given in Figure 2 described
19 hereinafter.

20 For a scrubbing solution comprising about 30 wt.%
21 K_2CO_3 and cooled to 160°- 130°F little or none of the other
22 components come out of the solution. If this 30 wt.% K_2CO_3
23 solution is cooled to 100°F and below, then $KHCO_3$ will also
24 precipitate from the solution and the process will no longer
25 be selective. Similarly, for a solution comprising 25 wt.%
26 K_2CO_3 and cooled to a temperature between 80° and 140°F only
27 cyclic urea is precipitated from the solution.

28 The invention disclosed herein describes the temper-
29 ature region in which the cyclic urea can be selectively
30 precipitated and removed without removing any of the other
31 many components in the solution.

32 BRIEF DESCRIPTION OF THE DRAWINGS

33 Figure 1 is a schematic flow diagram of one embodi-
34 ment of the claimed invention.

35 Figure 2 is a plot of the typical operating region
36 in which cyclic urea can be selectively precipitated as a

1 function of wt.% K_2CO_3 in a solution and the solution temper-
2 ature.

3 DETAILED DESCRIPTION OF THE INVENTION

4 The acidic components which will be removed from
5 the gaseous mixture by the scrubbing process will preferably
6 be selected from the group consisting of CO_2 alone or in
7 combination with H_2S , SO_2 , CS_2 , HCN , COS and the oxygen and
8 sulfur derivatives of C_1-C_4 hydrocarbons.

9 The alkaline material comprising basic alkali salts
10 or metal hydroxides will be selected from the group consist-
11 ing of alkali metal bicarbonates, carbonates, hydroxides,
12 borates, phosphates and their mixtures.

13 The contacting of the absorbent mixture and the acid
14 gas may take place in any suitable contacting tower. In such
15 processes, the gaseous mixture from which the acid gases are
16 to be removed may be brought into intimate contact with the
17 absorbing solution using conventional control means such as
18 a tower packed with, for example, ceramic rings or with bub-
19 ble capped plates or sieve plates or a bubble reactor. In a
20 preferred mode of practicing the invention, the absorption
21 step is conducted by feeding the gaseous mixture into the
22 base of the tower while the lean absorbing solution is fed
23 into the top. The gaseous mixture, free largely from acid
24 gases, emerges from the top. Preferably, the temperature
25 of the absorbing solution during the absorption step is in
26 the range from about 150°F to about 270°F and more preferably
27 from 150°F to about 250°F. Pressures may vary widely, accept-
28 able pressures being between 5 and 2000 psig. In the desor-
29 ber, the pressures will range from about 1 to 15 psig. The
30 process can be better understood by reference to the follow-
31 ing detailed description.

32 Referring to the figure, sour gas is introduced via
33 line 1 into absorption column 2 where it is contacted with
34 the aqueous scrubbing solution introduced via line 22. The
35 scrubbing solution is at a temperature of about 200°F and has
36 an amine concentration of from 1 to 10 wt.%, preferably 3 to

1 8 wt.%. As the absorbent liquid passes down the absorber
2 column, acid gas impurities are absorbed.

3 The absorbent solution, enriched with acid gas im-
4 purities, passes out of absorber column 2 into line 3 which
5 passes the enriched solution through heat exchanger 4 into
6 line 5. Line 5 passes the enriched solution into the regen-
7 erator 6 where the acid gases are stripped from the solution
8 and pass overhead through line 11. The lean solution formed
9 in the regenerator column passes to the bottom of column 6
10 and out via line 7 which feeds the lean solution to reboiler
11 8 where it is boiled by steam entering via line 9, the acid
12 vapors being passed via line 10 to the regenerator 6. The
13 lean (essentially acid free) solution passes out of reboiler
14 8 via line 12. Line 12 passes the lean solution through
15 pump 13 into line 14. Line 14 passes the lean solution into
16 heat exchanger 4 and some of the solution is cooled further
17 in heat exchanger 19 with cooling water entering via line 18
18 down to cool the solution to the temperature range in which
19 substantially only cyclic urea is precipitated. As used
20 herein, the term "precipitate" is defined as particles which
21 are separated from the solution irrespective of whether the
22 particles would settle to the bottom of the solution or float
23 on top. In this process the cyclic urea removed from the
24 solution is less dense than the solution and hence will float
25 on the surface. For a solution comprising about 30 wt.-%
26 K_2CO_3 the solution should be preferably cooled to 180°F to
27 120°F, as indicated in Figure 2, more preferably to 160°
28 to 130°F, most preferably 150° to 135°F to selectively pre-
29 cipitate the cyclic urea degradation product while keeping
30 all other components in the solution. Similarly, as shown in
31 Figure 2, a 25 wt.-% K_2CO_3 solution should be cooled to a
32 temperature ranging between 80°F and 140°F, preferably 90°-
33 120°F, to selectively precipitate only the cyclic urea.
34 Maximum cooling effect for typical K_2CO_3 concentrations may
35 be obtained by operation at a temperature in the range of
36 between 120°F-80°F. The solution containing the cyclic urea

1 solids is passed into line 15 which passes it to filter ele-
2 ment 16. The cyclic urea degradation product accumulates on
3 the filter and is thereby selectively removed from the lean
4 solution. When the ultimate buildup of the insoluble mater-
5 ial on the filter causes a pressure drop of, for example,
6 about 25 psi, a secondary filter 17 is cut in to allow con-
7 tinued operation of the process during the cleaning of the
8 segregated filter.

9 The lean solution passes out of filter 16 via line
10 23 and joins with some of the other lean solution in line 21
11 which was not cooled by exchanger 19. These two streams
12 combine and enter the top of the absorber. The purified
13 gas passes out of absorber 2 via line 24. Hot water having
14 a temperature of about 230°F is passed via line 20 into
15 filter element 16 in order to wash the cyclic urea from the
16 filter and to permit its effective reuse.

17 DESCRIPTION OF THE PREFERRED EMBODIMENTS

18 The following is a summary of several examples
19 which describe the invention.

20 EXAMPLE 1

21 A gas treating solution was prepared which had the
22 following composition: 30 wt.% K_2CO_3 (with 10% as $KHCO_3$).
23 6.0 wt.% cyclohexyl-1,3-propane diamine, 6.1 wt.% pipercolinic
24 acid, 56.2 wt.% water, and 1.7 wt.% cyclic urea, i.e.,
25 1-cyclohexyl-hexahydro-2-pyrimidinone. The solution at 200°F
26 was present as a single liquid phase. This solution was then
27 cooled at 150°F and was passed over a filter element which
28 was a cotton wound element having a nominal 100 μ size. The
29 cyclic urea content was decreased from 1.7 wt.% to less than
30 1.0 wt.%. The filtration rate was 0.5 gpm and the filter
31 size was a 3" diameter cylinder, 4" high with an inner opening
32 of about 1" diameter. The filter cake was analyzed to be
33 pure cyclic urea with none of the other solution components
34 present. This example shows that the cyclic urea can be
35 selectively removed by this process. It is not necessary to
36 completely remove all the cyclic urea but just to be able

1 to keep the concentration at a nominally low level even though
2 it is constantly being produced within the process.

3 EXAMPLE 2

4 The gas treating solution described in Example 1
5 was cooled to 130°F at which point considerably more cyclic
6 urea came out of the solution. Operation of the process at
7 these conditions, however, produced a somewhat inoperable
8 condition due to the plugging tendency of the resulting pre-
9 cipitate. In the flowing system in which the stream is being
10 continuously filtered such a line plugging tendency as
11 observed above made the system somewhat inoperable. Therefore,
12 this test indicated that for this solution cooling to below
13 130°F leads to an undesired operability problem. The solids
14 which were filtered during the test were again found to be
15 pure cyclic urea with none of the other components of the
16 solution present.

17 EXAMPLE 3

18 Another gas treating solution was prepared that was
19 similar to that shown in Example 1 but which had about 30% of
20 the K_2CO_3 present as $KHCO_3$. In this solution, cooling from
21 200°F to 150°F again caused the cyclic urea to selectively
22 precipitate from the solution. As the solution was further
23 cooled to somewhat below 100°F, it was found that the pro-
24 cess was no longer selective in that in addition to the
25 cyclic urea precipitating out, $KHCO_3$ was also precipitated
26 out. This test further shows the critical temperature range
27 over which the cyclic urea selectively precipitates. If the
28 temperature goes below about 100°F, the process is no longer
29 selective. The filter cake in this test was found to contain
30 substantial amounts of $KHCO_3$.

31 EXAMPLE 4

32 Another gas treating solution was prepared which
33 had a composition similar to that shown in Example 1 except
34 that the cyclic urea content was 1.4 wt.%. This solution
35 was cooled to 148°F and was filtered using a filter arrange-
36 ment as described in Example 1 but with a 5 μ element rather

1 than the 100μ element. The run was carried out at 0.5 gpm
2 and the filter cake collected was analyzed and was found to
3 be pure cyclic urea. In this test, the run was carried out
4 until the pressure drop across the element reached about 25
5 psi.

6 EXAMPLE 5

7 After completion of the run described in Example 4
8 the filter had a pressure drop of 25 psi due to the cyclic
9 urea cake on the filter element. This cake was washed with
10 170°F water and a second cycle was attempted in which case
11 a run of only about 25% as long as the initial run occurred.
12 This indicated that the washing with 170°F water was not an
13 effective cleaning process. The filter was then washed with
14 230°F water and then another cycle was attempted. In this
15 case, the cycle length was equivalent to that of the initial
16 cycle on the fresh filter. Three more complete cycles were
17 run in which the filter cake was washed with 230°F water
18 after a 25 psi pressure drop had built up due to the accumu-
19 lation of the cyclic urea cake. In each case the amount of
20 cyclic urea removed was the same as that with a new filter.
21 These tests indicated an effective washing technique in which
22 the water temperature criticality was demonstrated.

23 EXAMPLE 6

24 Another gas treating solution was prepared compris-
25 ing: 25 wt.% K_2CO_3 (with 20% of this present as KHCO_3), 6.3
26 wt.% cyclohexyl-1,3-propane diamine, 3 wt.% pipecolinic acid,
27 0.8 wt.% cyclic urea and the balance water. The solution was
28 then cooled to about 85°F . The filter cake was collected,
29 analyzed, and found to be substantially pure cyclic urea.

30 Although the subject process has been described with
31 reference to a specific embodiment, it will be understood that
32 it is capable of further modification. Any variations, uses
33 or adaptations of the invention following, in general, the
34 principles of the invention are intended to be covered,
35 including such departures from the present disclosure as come
36 within known or customary practice in the art to which the

- 12 -

1 invention pertains and as may be applied to the essential
2 features hereinbefore set forth, and as fall within the scope
3 of the invention.

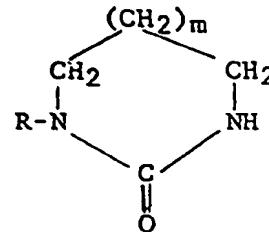
THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. An acid gas scrubbing process providing for the selective removal of a cyclic urea reaction product which forms as a by-product of the acid gas removal, said process comprising:

a. contacting an acid gas mixture with an aqueous solution in an absorption zone, said aqueous solution comprising a basic alkali salt, or metal hydroxide selected from the group consisting of alkali metal bicarbonates, carbonates, hydroxides, borates, phosphates and their mixtures, and an activator for said basic salt comprising at least one sterically hindered amine having the generic formula:



where R is a secondary or tertiary alkyl or cycloalkyl hydrocarbon having 4 - 20 carbon atoms and m is 2 - 5, at elevated temperatures and pressures such that there is formed a cyclic urea reaction product having the formula:



where R is a secondary or tertiary alkyl or cycloalkyl hydrocarbon having 4 - 20 carbon atoms and m is 0 - 3;

b. passing said acid gas rich aqueous solution to a regeneration zone where it is contacted with steam to remove the acid gas impurities therefrom;

c. cooling a portion of the lean solution exiting

from said regeneration zone to a temperature ranging from 180 - 80°F. to selectively precipitate the cyclic urea from said lean solution;

d. passing said lean solution containing said precipitated cyclic urea to a separation zone to remove at least a portion of the cyclic urea from said lean solution.

2. The process of claim 1 wherein said sterically hindered amine is an N-alkyl alkylene diamine.

3. The process of claim 2 wherein said lean solution exiting from said regeneration zone is cooled to temperatures ranging from 130° - 160°F.

4. The process of claim 3 wherein said sterically hindered amine is cyclohexyl-1,3-propane diamine.

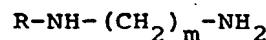
5. The process of claim 4 wherein said lean solution exiting from said regeneration zone is cooled to temperatures ranging from 135° - 150°F.

6. The process of claim 5 wherein said cyclic urea is removed by passing said lean solution through separation media comprising porous filters or activated carbon beds.

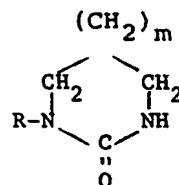
7. The process of claim 5 wherein the accumulation of said cyclic urea on said filter is monitored until the pressure drop across said filter reaches about 25 psi at which time said filter is segregated and a second filter is substituted therefor.

8. An acid gas scrubbing process providing for the selective removal of a cyclic urea reaction product which forms as a by-product of the acid gas removal, said process comprising:

a. contacting an acid gas mixture with an aqueous solution in an absorption zone, said aqueous solution comprising a basic alkali salt, or metal hydroxide selected from the group consisting of alkali metal bicarbonates, carbonates, hydroxides, borates, phosphates and their mixtures, and an activator for said basic salt comprising at least one sterically hindered amine having the generic formula:



where R is a secondary or tertiary alkyl or cycloalkyl hydrocarbon having 4 - 20 carbon atoms and m is 2 - 5, at elevated temperatures and pressures such that there is formed a cyclic urea reaction product having the formula:



where R is a secondary or tertiary alkyl or cycloalkyl hydrocarbon having 4 - 20 carbon atoms and m is 0 - 3;

b. passing said acid gas rich aqueous solution to a regeneration zone where it is contacted with steam to remove the acid gas impurities therefrom;

c. cooling a portion of the lean solution exiting from said regeneration zone to a temperature ranging between 120° - 80°F. to selectively precipitate the cyclic urea from said

lean solution;

d. passing said lean solution containing said precipitated cyclic urea to a separation zone to remove at least a portion of the cyclic urea from said lean solution.

9. The process of claim 8 wherein said sterically hindered amine is an N-alkyl alkylene diamine.

10. The process of claim 9 wherein said lean solution exiting from said regeneration zone is cooled to temperatures ranging between 90° - 120°F.

11. The process of claim 10 wherein said sterically hindered amine is cyclohexyl-1,3-propane diamine.

12. The process of claim 11 wherein said cyclic urea is removed by passing said lean solution through separation media comprising porous filters or activated carbon beds.

13. The process of claim 11 wherein the accumulation of said cyclic urea on said filter is monitored until the pressure drop across said filter reaches about 25 psi at which time said filter is segregated and a second filter is substituted therefor.

14. The process of claim 1 or 8 wherein a loading of about 1 to about 10 SCF of acid gas per gallon of said aqueous solution is achieved in step (a).

15. The process of claim 1 or 8 wherein the regeneration zone in step (b) is operated at temperatures ranging from about 200°F to about 250°F and pressures ranging from about 1 psig to about 15 psig.



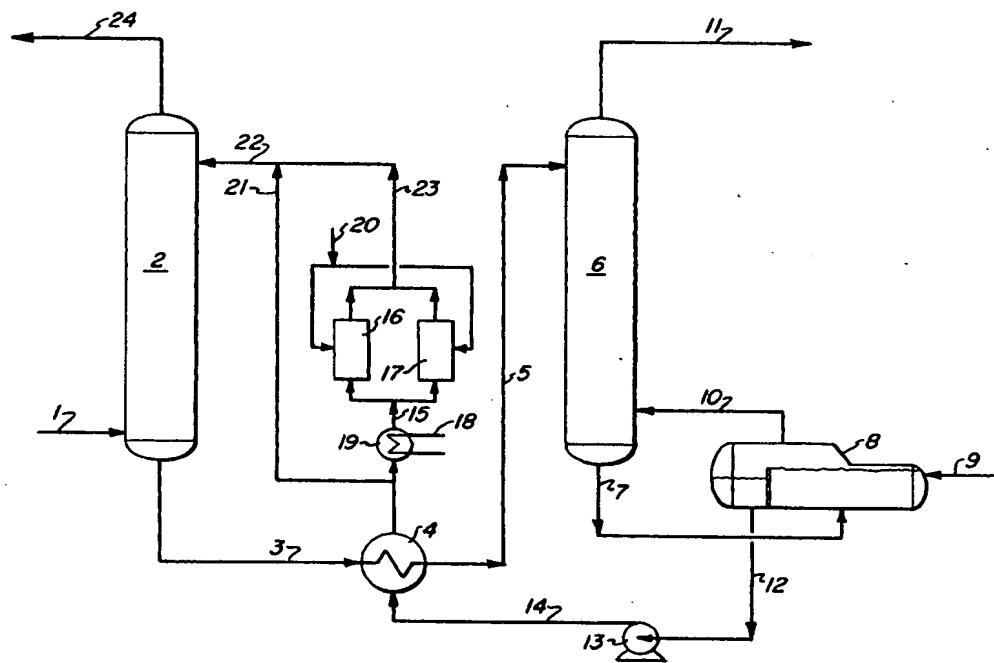
ABSTRACT OF THE DISCLOSURE

In the process of the present invention, a cyclic urea degradation product formed as a by-product of a hindered amine acid gas scrubbing process is removed from a circulating amine scrubbing solution by employing a selective precipitation of the cyclic urea followed by filtration. The selective precipitation is carried out by cooling the circulating solution to a particular temperature level such that the cyclic urea comes out of solution while the other components remain in solution.

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FIG. 1

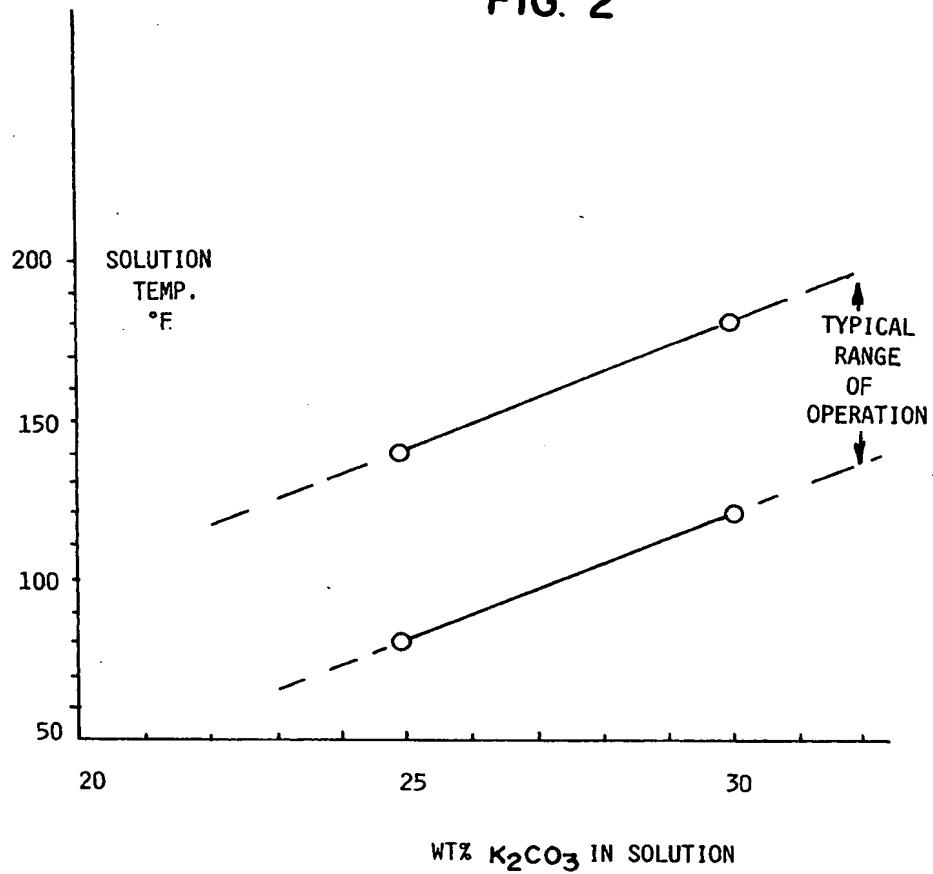


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FIG. 2



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